A study of chemically and biologically synthesized hematite phase co-doped ferric oxide (Fe_2O_3) nanoparticles

SYNOPSIS

Submitted in partial fulfilment of the requirements for the award of the degree of
DOCTOR OF PHILOSOPHY
in
PHYSICS

Submitted by

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2021-2022

1. Introduction

1.1. Nanotechnology

The Nobel Prize-winning physicist Richard Feynman described a method to manipulate individual atoms and molecules (Feynman, 1960). Professor Norio Taniguchi first used the word "nanotechnology" in 1974, defining it as the processing, separation, consolidation, and deformation of materials by a single atom or molecule. Nanotechnology is a new interdisciplinary field that combines chemistry and physics principles with mechanical design, structural analysis, computer science, electrical engineering, and system engineering principles. It enables the creation of a wide range of materials, tools and goods with capabilities not previously possible. Understanding and manipulating matter at a scale of 1 to 100 nanometers is known as nanotechnology (Kiara & Dabral, 2013). In general, nanotechnology focuses on the development of materials and/or devices that are at least 100 nanometers or below in size in at least one dimension.

1.2. Nanoparticles (NPs)

According to the phrase, NPs range in size from 1 to 100 nm, can be made of inorganic or organic materials, and contain several unique features when compared to bulk materials. An ultrafine particle known as a nanoparticle has size measured in nanometers (1 nm = 10^{-9} meters). In addition to being produced by human activity, NPs also occur in the natural world. Manufactured NPs may have uses in a number of fields, including medical, engineering, catalysis, and environmental remediation. This is because they have unique material properties due to their submicroscopic size (Wu, He & Jiang, 2008).

1.3. Iron Oxide (Fe_2O_3)

An inorganic compound with the formula Fe_2O_3 is known as iron (III) oxide. Hematite and red iron oxide are other names for it. Rocks from diverse times contain this chemical. It appears to be a constant shade of red-brown. The pH of it is 7. This can be produced in a laboratory by electrolyzing a sodium bicarbonate solution with an inert electrolyte, an iron anode. The hydrated iron (III) oxide dehydrates at a temperature of about 400 °C. There are numerous polymorphs of Fe_2O_3. Four oxygen ligands in tetrahedral locations are coupled to some of the Fe in the polymorph (Muhajir, Puspitasari & Razak, 2010).

1.4. Hematite (α-Fe_2O_3)

The most common is α-Fe_2O_3, which has a corundum rhombohedral structure. Nature contains the mineral hematite, which is used to extract primary iron ore. It is

antiferromagnetic below 260 K (Morin transition temperature), and mildly ferromagnetic between 260 K and 950 K (Tadic et al., 2014). Utilizing both liquid phase precipitation and thermal breakdown makes it simple to create. Pressure, particle size, and magnetic field intensity are only a few of the variables that affect its magnetic properties. Hematite (α-Fe_2O_3) is frequently used in catalysts, pigments, and gas sensors due to its inexpensive cost and great corrosion resistance as one of the most stable iron oxide and n-type semiconductors under atmospheric temperatures. It can also be used as a raw material to create the minerals magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3), which have drawn a lot of attention in recent years for their potential use in both fundamental science and technology (Colombo et al., 2015).

1.5 *Azadirachta indica*

Azadirachta indica (Neem), often known as "Indian Lilac" or "Margosa," is a member of the tribe Melieae and the families Meliaceae and Meloideae (**Figure 1**). Many diverse societies have employed *Azadirachta indica* medicinally throughout history. The exudates of the *Azadirachta indica* plant contain numerous substances that have been utilized by people as medicines. The Meliaceae family includes neem. Fast-growing evergreen *Azadirachta indica* trees are widely distributed in India, Africa, and America. Due to its therapeutic qualities, it has been utilized in Ayurvedic medicine for more than 4000 years (Upadhayay & Vigyan, 2014).

Figure 1: *Azadirachta indica* plant

It is common to use plants or isolated products of them to treat or serve as anti-inflammatory agents. Neem seed kernel powder had antidiabetic and antihyperlipidemic benefits on alloxan-diabetic rabbits (Uchegbu et al., 2011). Neem leaves were examined for their antibacterial, antisecretory, and antihemorrhagic properties against Vibrio cholera using a methanolic extract. Using the agar dilution technique, the aqueous and ethanolic

extracts of *Azadirachta indica* leaves were shown to have antidermatophytic activity against dermatophytes from 88 clinical isolates (Al Akeel, et al., 2017).

1.6. Doping

Doping NPs is not as straight forward as doping macroparticles. It is entirely possible that the doping element prefers to concentrate on the surface (and the surface in NPs, as is known, plays a leading role). Doping, on the other hand, can result in saturation of the volume with the doping element, with only a minor effect on the surface (Chen et al., 2011). In general, if a substance's degrees of freedom are doubled (for example, the surface of "FeO" NPs obviously contains an excess of oxygen in normal cases (we live in an oxygen world), it is better to describe it as Fe_3O_4 (or even Fe_2O_3), then a similar phenomenon should be taken into account when doping of course, the assumption of uniform doping element distribution. Metal oxide doping is commonly pursued to improve electronic, optical, and catalytic performance (Garcia et al., 2011). The primary motivation for such property tuning is to gain control over doping, which alters electron transfer characteristics through changes in the crystalline structure of the pristine metal oxide NPs (Lounis et al., 2014).

2. Literature Review

Satheesh et al. (2019) were prepared the UV-vis DRS, FT-IR, XRD, SEM, EDX, and VSM techniques were used to characterize the different metal (M = Cu, Ni, and Co) doped iron oxide (α-Fe_2O_3) NPs that were created. The findings showed that Cu-Fe_2O_3 had greater photocatalytic activity than Fe_2O_3, Ni-Fe_2O_3, and Co-Fe_2O_3

Sharma et al. (2021) published their paper entitled "Thermal catalytic activity of α-Fe_2O_3 hexagonal cones produced from *Azadirachta indica* leaf extract" The production of -HNPs was indicated by an absorption peak in the UV-Vis spectra at about 565 nm; the energy band gap was computed and found to be 2.19 eV. The XRD results revealed that the average crystallite size was approximately 38.2 nm, and the crystal system was determined to be compatible with the corresponding rhombohedral phase of hematite NPs with crystalline nature. FESEM study with an average diameter of 400-500 nm indicated a hexagonal cone-like.

Helan et al. (2022) utilized the Nickel oxide NPs (NiO-NPs) were created using a biosynthesis method and phytoconstituents found in neem leaves. Morphological studies using Transmission Electron Microscope (TEM) reveals that the biosynthesized NiO-NPs were in shape of oblong with 12 nm in size Finally, agar diffusion assays were used to test

the NiO-NPs for bacterial activity against *Staphylococcus aureus* (MTCC 1430) and *Escherichia coli* (MTCC 739.

Lassoued et al. (2018) synthesized the chemical co-precipitation method was used to synthesis, characterize and apply pure and Ni-doped α-Fe_2O_3 (hematite) NPs for photocatalytic applications. UV-Vis absorption measurements confirm that the decrease in particle size is accompanied by a decrease in band gap value, which drops from 2.02 eV for α-Fe_2O_3 to 1.81 eV for 8 mol % Ni-doped α-Fe_2O_3. The photocatalytic activity of Ni-doped α-Fe_2O_3 particles, on the other hand, was investigated using methylene blue (MB) as a model organic pollutant. After 140 minutes of irradiation with 8 mol % Ni-doped α-Fe_2O_3 NPs, the discoloration of MB reached 86 %

Nagar and Devra (2022) prepared the *Azadirachta indica* leaf broth was used to create copper NPs, and the effects of several reaction parameters on the production rate and morphology of the copper NPs, including temperature and pH of the medium and precursor salt content, leaf broth were examined. Different objective approaches were used to characterize the biosynthesized copper NPs, and the results showed that the particles are crystalline, cubical in shape, on average 48 nm in size, and extremely stable.

Jacob and Renuga (2018) study focus on the structural and magnetic characterization of zinc substituted hematite NPs synthesized via co-precipitation, as well as the antimicrobial and anti-diabetic activity of these NPs. Rhombohedral in structure, the produced Zn-Fe_2O_3 NPs are in the hematite phase. By using the disc diffusion and pancreatic technique, the antibacterial and antidiabetic activities of synthesized Zn-Fe_2O_3 NPs are examined, and it reveals a high level of inhibition.

Lubis & Al-Haitami (2021) prepared the iron ore was used as the hematite source in the impregnation procedure to create copper-doped hematite (Cu-doped α-Fe_2O_3) NPs. The prepared samples morphological and structural characteristics were investigated. The hematite that was recovered from iron ore was in the hematite phase, according to the XRD data. Copper-doped hematite had an average crystallite size that was a little smaller than that of hematite. Under UV and solar light irradiation, the copper-doped hematite NPs shown high photocatalytic activity on degradation of indigo carmine (IC) dye, with the degradation percentage being 98.21 and 99.78 %, respectively

Mansour, Omri and Ammar (2019) synthesized coprecipitation process, pure α-Fe_2O_3 and co-doped α-Fe_2O_3 (CFO) NPs were created. The findings, which are in line with those of Raman spectroscopy, demonstrated that CFO NPs have a rhombohedral structure and that the Co^{2+} ions were effectively incorporated into the α-Fe_2O_3 crystal lattice. The

results from TEM and SEM images are compatible with the crystallite sizes, which were in the 36-38 nm range.

Chahal, Kumar and Kumar (2020) synthesized Zinc (Zn) doped hematite (α-Fe_2O_3) NPs with varying concentrations (pure, 2, 4 and 6 %) were synthesized using the sol-gel method. The effect of divalent Zn ions on the structural, optical, and dielectric properties of hematite was investigated. The X-ray diffraction (XRD) pattern of synthesized samples was indexed to the rhombohedral R3c space group of hematite with crystallite sizes ranging from 14 to 21 nm. Zn^{2+} doped hematite is an efficient material with potential applications in photocatalysis, and its higher dielectric constant at room temperature makes it suitable for high energy storage devices.

Yilleng et al. (2020) utilized metal (iron and copper) NPs were created using an extract from neem leaves. UV-vis spectrophotometer analysis was used to track the emergence and stability of the reduced metal NPs in the colloidal solution. Cu NPs and Fe NPs have peaks that are spherical and dendrimer, respectively and have particle sizes between 250 nm and 550 nm and 250 nm and 350 nm, respectively. Cu NPs has a 20 mm ±1 zone of inhibition on gram-negative bacteria like *Staphylococcus aureus* and a 12.5 mm ±1 zone of inhibition on gram-positive bacteria like *Escherichia coli*.

Ajayan and Hebsur (2020) prepared Neem (*Azadirachta indica*) and tulsi (*Ocimum tenuiflorum*) leaf extracts were used in a study to create zinc oxide NPs. they produced zinc oxide NPs absorbance peaks were found in the UV wavelength region (359 and 364 nm for neem and tulsi, respectively). Zinc oxide NPs made from neem and tulsi had average diameters of 101.6 and 122.4 nm, respectively, according to a particle size analyzer (PSA). ZnO NPs made from neem showed flakes or flower-like shape in scanning electron microscopy (SEM), with sizes between 100 and 300 nm

Paulson and Jothibas (2021) synthesized Sol-gel synthesis was used to create hematite (α-Fe_2O_3) NPs at a reasonable price. Shape, size and particle homogeneity were all guaranteed with the introduction of temperature, according to the surface morphological study. The phase purity of the produced NPs is confirmed by FT-IR. The production of red-colored α-Fe_2O_3 C-2 NPs and the optical bandgap values of 1.92-1.97eV are confirmed by the optical absorption at 534 nm. The magnetic transition of NPs paramagnetic to weak ferromagnetic properties was seen as the magnetometry steady.

Malathy and Revathi (2021) utilized *Cadiospermum halicacebium* leaf extract was used to create cobalt NPs in an economical and environmentally friendly way. As a reducing and capping agent, the leaf extract works. Several methods, including UV-visible

spectrophotometry, Fourier transform infrared spectrometry, XRD and scanning electron microscopy, were used to characterize the biosynthesized cobalt NPs. Ethanolic Extract was used to make cobalt NPs in an economical and environmentally friendly way, and several methods were used to describe them. Cobalt NPs were proved to have formed using spectroscopic methods, and microscopic analysis verified their spherical shape and average particle size of 48 nm. The disc diffusion method was used to assess the antibacterial activity of the produced NPs. When compared to the antibiotic Ciprofloxacin, the cobalt NPs demonstrated effective antibacterial action against the human pathogenic bacteria *S. Dysenteriae* and *E. faecalis.*

Lassoued (2021) synthesized optical, magnetic and photocatalytic characteristics of Zn-doped and undoped α-Fe_2O_3 NPs. The molar ratio of zinc precursors was altered during the simple co-precipitation process used to create the Zn doped hematite NPs. In the photodegradation of methyl orange, zinc doped α-Fe_2O_3 displayed improved photocatalytic activities (MO). The maximum photocatalytic activity was demonstrated by the 6 % Zn-doped α-Fe_2O_3 NPs, which also nearly completely discolored MO solutions.

Grabsi, et al. (2022) utilized successfully synthesized using the sol-gel method, two series samples of iron oxide NPs doped with nickel and cobalt had different doping values (x = 0.01; 0.03; 0.05 and 0.07), and they were characterized using X-ray diffraction, scanning electron, and vibrating sample magnetometer (VSM). Two series samples were subjected to X-ray diffraction analysis, which revealed the synthesis of α-Fe_2O_3 NPs along with $CoFe_2O_4$ and $NiFe_2O_4$ iron spinels. Along with the ferromagnetic behaviour of the two series doped with cobalt and nickel, magnetic measurements made using VSM revealed important magnetic properties as coercivity and magnetization.

3. Research Gap Identification

In fact, many researchers have studied synthesis of α-Fe_2O_3 and doped α-Fe_2O_3 NPs by using different types of transition elements but there is very less literature on the synthesis of co-doped α-Fe_2O_3 NPs by using transition elements as well as *Azadirachta indica* plant at particular temperature. The present study will discuss the comparative physical and chemical properties of anatase phase of chemical and green synthesized co-doped α-Fe_2O_3 NPs. In addition to this, synthesized NPs will be used for the photocatalytic and antioxidant activity.

4. Objectives

In the light of above discussions, the objectives of the present study are as follows:

- To study chemical and green synthesis of Hematite phase of co-doped α-Fe_2O_3 NPs with different transition elements through co-precipitation method.
- To characterize the synthesized co-doped α-Fe_2O_3 NPs by using different characterization techniques like X-ray diffraction (XRD), Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), UV-visible spectroscopy, scanning electron microscope (SEM), Transmission electron microscope (TEM) and Vibrating-sample magnetometer (VSM).
- To study the photocatalytic and antioxidant applications of synthesized NPs.

5. Proposed Methodology

5.1. Materials

Iron (III) chloride hexahydrate (98 %, Sigma Aldrich), Polyvinylpyrrolidone (PVP-1,300,000), Cobalt chloride hexahydrate (98.5 %, Sigma-Aldrich), Nickle (II) chloride hexahydrate (97 %, Sigma-Aldrich), Copper (II) chloride dihydrate (98.5 %, Sigma-Aldrich), Zinc chloride (98 %, Sigma-Aldrich), Sodium hydroxide (98 %, Sigma-Aldrich) distilled water and 2,2-diphenyl-2-picrylhydrazyl hydrate (DPPH) are the chemicals used in this experimental work. Plant leaves of *Azadirachta indica* will be collected from local areas of district Hamirpur, Himachal Pradesh for the preparation of extract.

5.2. Synthesis of *Azadirachta indica* plant extract

The fresh leaves of *Azadirachta indica* plant will be collected from local areas of district Hamirpur, Himachal Pradesh. The collected leaves will be washed properly with tap water and then with distilled water to remove the dust, microorganism, any particles or other impurities that may be on the leaves. Washed leaves will be weight accurately 10 gm. The weight leaves are to be taken in the mortar piston and was chopped and crushed well. Further, well-minced leaves will be boiled at temperature 70 ^oC for about 30-35 minutes in 100 ml distilled water. The assembly will consist of the attachment of the condenser with the round bottom flask standing on the rota-mantle with constant stirring. The boiled extract will be cooled and filtered through Whatman filter paper. The filtered extract solution will be used for the synthesis of co-doped α-Fe_2O_3 NPs (**Figure 2**).

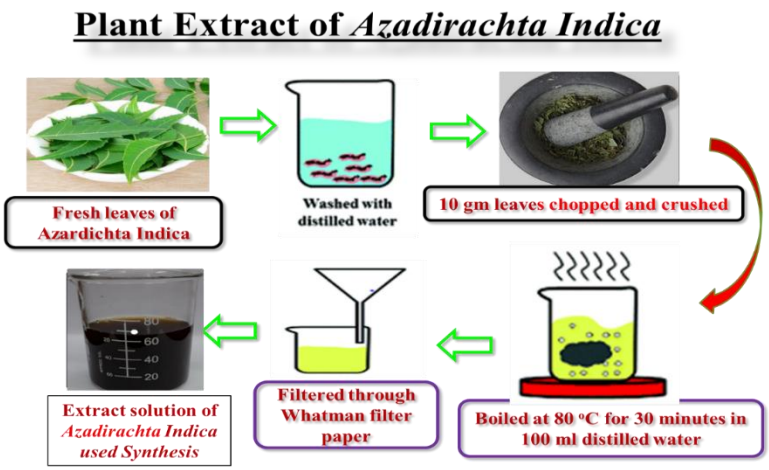
Plant Extract of Azadirachta Indica
Fresh leaves of Azardichta Indica
Washed with distilled water
10 gm leaves chopped and crushed
Boiled at 80 °C for 30 minutes in 100 ml distilled water
Filtered through Whatman filter paper
Extract solution of Azadirachta Indica used Synthesis

Figure 2: Plant extract preparation of *Azadirachta indica.*

5.3. Synthesis of co-doped α-Fe_2O_3 NPs

Sure, here's a reframed version: The synthesis of co-doped α-Fe_2O_3 NPs will involve the co-precipitation method. Initially, we'll utilize specific precursors, and for the synthesis process, a combination of PVP or plant extract will serve as the surfactant. To begin, the precursors will be dissolved in distilled water. Paired metal salts, each at desired molar concentrations, will be dissolved separately in distilled water. Then, they will be gradually added dropwise into the aqueous solution, maintaining constant stirring using a magnetic stirrer at room temperature. Subsequently, PVP or plant extract will be dissolved in distilled water and added slowly to the solution. The pH of the solution will be adjusted as necessary. The mixture will undergo stirring for several hours at room temperature. Following this, the resulting nanoparticles will undergo multiple washes using distilled water and ethanol. They will then be dried in a vacuum oven for several hours. After the drying process, the co-doped α-Fe_2O_3 NPs will be subjected to calcination at various temperatures.

5.4. Characterization of synthesized co-doped α-Fe_2O_3 NPs

The synthesized co-doped α-Fe_2O_3 NPs will be characterized to determine its various physical as well as chemical properties such as crystallite size, crystal structure, lattice parameters, crystal phase, bandgap, functional groups, surface morphology, particle size and also magnetic properties. The characterization techniques will be used for determining these parameters are X-ray diffraction (XRD), Raman spectroscopy, UV-visible spectroscopy, Fourier transform infrared spectroscopy (FTIR), Scanning electron microscope (SEM), Transmission electron microscope (TEM) and Vibrating-sample magnetometer (VSM).

5.5. Photocatalytic activity

In this study, experiments will be performed to evaluate the photocatalytic properties of synthesized co-doped α-Fe_2O_3 NPs by photocatalytic degradation of commercial dye. The photocatalytic decomposition of dyes will be examined by measuring the absorbance at regular time intervals by using UV-Visible spectrophotometer.

5.6. Antioxidant activity

The antioxidant activity of synthesized α-Fe_2O_3 NPs will be examined by investigating the radical scavenging ability against 2,2-dipenyl-1-phenylhydrazyl (DPPH) free radical at different concentration. The absorbance of solutions will be measured by UV-Visible spectrophotometer.

6. Thesis Outline

The thesis will be comprised of following chapters:

Chapter 1 (Introduction): It will constitute of the brief account about nanotechnology, nanoparticles polymer PVP, plant *Azadirachta indica* and applications of α-Fe_2O_3 NPs. It will also include the objectives of proposed study.

Chapter 2 (Literature Review): This chapter will deal with intense literature with context to the synthesis, characterization and applications of α-Fe_2O_3 NPs. It will also include the research gap identification.

Chapter 3 (Materials and Methods): This chapter will comprise the detail of materials, chemical as well as green synthesis, characterization techniques and applications of co-doped α-Fe_2O_3 NPs.

Chapter 4 (Results and Discussion): This chapter will include the reports regarding physical and chemical properties of anatase phase of co-doped α-Fe_2O_3 NPs along with analysis and comparison of the findings the work.

Chapter 5 (Summary and Conclusion): It will include the overall summary and conclusion of the entire study with regard to the work with context to the objectives cited.

Chapter 6 (References): This will include references which will be strictly according to the Harvard style.

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CERTIFICATE
(SYNOPSIS APPROVAL CERTIFICATE)

This is to certify that the Synopsis for Ph.D. Thesis titled, **"A STUDY OF CHEMICALLY AND BIOLOGICALLY SYNTHESIZED HEMATITE PHASE CO-DOPED FERRIC OXIDE (Fe_2O_3) NANOPARTICLES"**, submitted by **Mr. Pankaj Kumar** to the Career Point University, Hamirpur (H.P.), India in partial fulfillment of the requirements for the degree of **DOCTOR OF PHILOSOPHY** in the Department of **Physics**, School of **Basic and Applied Sciences** has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated 27.06.2022.

Dr. Naveen Thakur, Professor
Department of Physics, CPU, Hamirpur (H.P.)
(Supervisor)

Research Advisory Committee

Dr. Indu Sharma, Associate Professor
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